FLOW MEASUREMENT DEVICE

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BACKGROUND

Related Application

This application is based on U. S. provisional patent application serial no. 60/396,216 entitled, Flow Measurement Device filed July 16, 2002.

Field of the Invention

The field of the invention generally relates to techniques for precisely measuring the flow rates of gases and liquids and mixtures thereof. In particular, the field of the invention relates to a flow measurement device positioned in a flow path for measuring liquids and gases, or a mixture thereof that achieves a previously unattainable degree of precision. Positioning of the flow measurement device in the flow path eliminates the complexity and inaccuracies inherent in a conventional bypass structure and enables precise real time control of process parameters.

Background of Related Art

Accurate flow rates of gases and liquids are of wide-ranging interest in our economy. In particular, as semiconductor feature sizes shrink to atomic scales, it is essential that mass flow parameters be maintained with tighter control than was previously possible. Sophisticated applications are common in semiconductor manufacturing, pharmaceuticals and optics among other areas of commerce. Small process variations, too small to be accommodated by conventional mass flow devices, can result in unacceptable process variations when feature sizes are measured with respect to nanometers.

Two concepts are commonly in use for sophisticated applications requiring precise mass flow rates. These two concepts are commonly referred to as "bypass or capillary" mass flow

meters (MFM) and "bubbler" mass flow metering devices. Although there are a number of variations of these conventional mass flow meters, they all share a number of problems and limitations. These problems and limitations include, inherent inaccuracies, inherent process variations, maintenance difficulties, cleaning difficulties and inefficient size.

The conventional "bypass or capillary" MFM (**Figure1**) makes use of the change in a temperature differential caused by mass flow through the bypass structure. Typical conventional MFM's of this design are disclosed in U.S. Patent No. 2,729,976 (Laub), U.S. Patent No. 3,938,384 (Blair) and U.S. Patent No. 4,487,062 (Olin). When the temperature differential (Temperature, T_2, greater than Temperature T_1) is measured by electronic temperature sensors, the flow signal becomes an electrical signal that is highly useful for automated process control. The MFM can be small, accurate and minimally contaminating to the process of interest.

However, the conventional "bypass or capillary" MFM has subtle limitations that increasingly limit its suitability for sophisticated processing. Heat transfer within the sensor itself changes due to many factors, such as intimacy of the heating element bond to electrical insulation, electrical insulation bond to capillary, heat transfer through capillary wall, deposits built up inside capillary, and heat transfer from edge to the center of capillary flow. These changes in heat transfer characteristics affect the correlation between heater energy and temperature differential and reduce the precision of the MFM.

The capillary part of this traditional MFM is a very small flow tube intended to minimize temperature difference from an edge to the center of flow. Such temperature difference is undesirable because it introduces an uncertainty in the amount of fluid being heated and thus an uncertainty in the amount of fluid flow. Typically, only a small portion of the total flow passes through the capillary. Thus, a large portion of the flow must pass through a calibrated by-pass without contributing to the flow measurement at all. An assumption must be made that flow proportions through the capillary and by-pass stay the same regardless of fluid composition, process temperature, process pressure and overall process flow rates. This assumption introduces an inherent inaccuracy into all "bypass or capillary" MFMs.

The above limitations apply to gaseous flow. For liquid flow, additional complications impose limitations on the accuracy of conventional methods of mass flow measurement. Bubbling, voiding or boiling can occur as the liquid passes through the capillary. This can result from increases in temperature associated with heating by the heater, pressure drops and directional changes, among other reasons, as the fluid passes into and through the capillary. These effects can result in a change in phase of liquid to vapor or the emergence of dissolved gases from the fluid. The introduction of two phases into the capillary significantly and

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unpredictably alters the correlation between heater energy and temperature differential. Under these circumstances, the flow signals become totally unreliable.

Because of conventional MFM difficulties in handling liquids, bubblers **(Figure 2)** are sometimes used as an alternative means of metering mass flow rates. Typical conventional bubbler designs are disclosed in U.S. Patent No. 4,134,514 (Schumacher), U.S. Patent No. 4,140,735 (Schumacher) and U.S. Patent No. 4,436,674 (McMenamin). Sophisticated applications require the use of gaseous ingredients to achieve the precise mass flow rates necessary in applications where atomic scales are important. Therefore, a mass flow metering device (bubbler) starting with a liquid ingredient requires a change of state; i.e. controlled evaporation to a gaseous state. The amount of liquid used is frequently very small which makes accurate measurement difficult. When the liquid is converted to a gas, a volume expansion of about 1000 times occurs which then makes measurement easier.

A bubbler, **(Figure 2)** is a container of liquid kept at a fixed temperature by the heater (with appropriate controls, enclosures, etc.). An inlet gas bubbles into the liquid, mixing with evaporant. Above the liquid level, the proportion of gas to evaporant is directly related to the liquid temperature and the container pressure. If container pressure surges higher, the evaporant proportion will be reduced. If container temperature is slightly cooler at the top, condensation will occur, also lowering evaporant proportion. If bubbling action is too vigorous, atomization can occur, producing aerosols that represent increased proportion. The liquid level must be kept constant or the thermal and evaporative characteristics of the bubbler change.

To keep a constant level as evaporation removes liquid, a level sensor must be provided. The level sensor signals additional liquid delivery through the refill port. Of course, the refill liquid must be the same temperature or the bubbler characteristics will change. If all goes well, a specific concentration of evaporant in inlet gas passes through the outlet to the process area. The outlet region must be kept sufficiently hot or condensation will occur, lowering evaporant concentration.

Obviously, accurate control of a bubbler is no simple task. For some processes, several liquids may be required. Each liquid requires its own bubbler. Even if the liquids can be freely mixed without altering their properties, the bubbling process constitutes distillation. Evaporation would distort the liquid proportions unless all liquids had exactly the same evaporation characteristics.

Fluid flow measurement is becoming critical to increasing operational efficiency of many commercial processes, such as in semiconductor process control. As semiconductor device feature sizes shrink to atomic dimensions, improved in line process measurements that do not

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introduce inaccuracies or assumptions are needed to control fluid flow. Therefore, what is needed is a new method for directly measuring fluid flow that provides precise *in situ* measurement of fluid flow rate for enabling real time control of process parameters without error.

SUMMARY

In accordance with the foregoing and other objectives, an aspect of the invention provides a flow measurement device that works equally well with gases, liquids or mixtures of gases and liquids. Another aspect of the invention provides greater control over flow rates with very high degree of precision. This is due to the fact that the measurement device eliminates the need for one or more bubblers. Accordingly, this eliminates bubbler pressure sensitivity and the bubbler requirement for exact (and unstable) gas vapor saturation.

An aspect of the present invention also eliminates the need for a capillary or bypass structure. Since the present measurement device is provided in the flowpath for direct measurement of flow, it advantageously eliminates the introduction of inaccuracies, bypass errors or inaccurate assumptions inherent in a conventional bypass structure.

Another aspect of the invention provides an internal electric heat source. Because the heat source is internal, the invention does not suffer from any of the heat transfer problems associated with conventional mass flow meters. Since the heat transfer associated with the heater forms the basis of flow measurement, improved accuracy is achieved.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows a conventional mass flow meter (MFM) including a capillary and bypass structure.

Figure 2 shows a conventional MFM including a bubbler.

Figure 3 shows a flow measurement device capable of measuring gases, liquids, or mixes thereof according to the present invention.

DETAILED DESCRIPTION

Referring to **Figure 3**, a new type of flow measurement device is provided that achieves improved measurement of gases, liquids, or mixtures thereof. When coupled with valves or other flow regulating equipment, the flow measurement device becomes a highly precise flow-metering device.

Heat flow always requires a temperature difference. In **Figure 1**, temperature T_1 and T_2, used for measurement, are not in the flow path. It can be expected that temperatures, in the flow path, will be different than T_1 and T_2, thus constituting an error. The capillary of **Figure 1** is a structural feature which minimizes this error, but then can only measure a small portion of the total flow. The capillary feature is more vulnerable to calibration changes caused by a change in heat flow characteristics from T_1 or T_2 to the total flow path. Debris is one such change. The intimacy of thermal contact between heater, insulation and capillary wall are other changes. These uncertainties are magnified when measuring liquids which degas and boil with severe disruption in heat transfer.

In contrast, the design concept of **Figure 3** eliminates these uncertainties, enabling the effective measurement of gases, liquids or mixtures thereof.

The flow measurement device **300** comprises an evaporator **308**, which may be a generally tubular structure. A cross section of the evaporator including the coaxial internal heater **302** is shown.

In operation, gas and liquid enter through gas and liquid lines **304** and **306**, respectively. Gas and liquid lines **304** and **306**define flow paths as indicated by arrows. Flow rate of the gas is assumed known (measured by any convenient device). The measurement can take place upstream in the flow paths prior to entering the flow measurement device **300**. Temperature of gas and liquid are measured by convenient devices giving temperatures at T_1 and T_2, respectively, located in the flow paths. The location where gas flow mixes with the liquid is chosen to ensure mixing of the gas and liquid phases and avoid phase stratification. This mixture enters the evaporator through a plenum or inlet at **307**. The evaporator **308** comprises the evaporator housing or shell **309**, a spiral channel **310** and heater **302**. Channel **310** comprises a single flow path defined by the longitudinal axis of the evaporator. The spiral channel is created by a series of diversion fins **311** which provide a means for inducing a spiral or helical flow path. The helical flow path ensures that unsuspected aerosols do not flow through the device without being heated.

The liquid in line **306** can be a mixture. Since all of the liquid is being evaporated by evaporator **308**, it doesn't matter if the components have different boiling points. Temperature at measurement point T_3 is located directly in the flow path of the gas and liquid evaporant mix as it exits the evaporator **308**. However, measurement point T_3 should be adjusted in operation to be above the boiling point of any component flowing into the device to be sure it is evaporated. Heat insulation **312** is disposed around the evaporator and heater. The heat insulation **312** serves to make sure that the only heat entering or leaving the system comes from the heater **302**, which is monitored electronically through heater leads **314**.

When the composition and proportions of the liquid are defined for the specific application, the specific heats and heats of vaporization to change them from T_2 to T_3 can be determined from physical property information sources. Given these compositions, proportions and physical properties, there is a precise thermal energy input from the heater (measured from heater leads **314**) necessary to increase the temperature to T_3 from an initial temperature of T_2. This precise thermal energy input is also directly related to liquid flow. Because thermal energy, T_3 and T_2 are measured, the liquid flow can be precisely determined based on a simple yet precise energy balance. Similarly, the gas flow (known) also will require a definite corresponding heat flow to raise temperature to T_3 from T_2 as a specified mass flow rate. The gas will not undergo a phase change; therefore heat of vaporization is not relevant.

Therefore, a precise mass flow rate measurement can be determined from the physical properties, measurement of heater energy and measurement of temperatures at T_3 and T_2. These flows, brought to temperature T_3 by a measured heat flow constitute a liquid flow rate measurement. If the heat input is kept constant, T_3 will change in proportion to gas and liquid flow also. However, because a change of state is involved, it would seem better to fix T_3 approximately above boiling and to vary heater power.

Thus, in an actual process, T_3 is chosen to suit the liquid used. The desired process will require a certain gas and liquid flow. Valves or other flow regulating equipment in the gas line sets this flow. Valves or other flow regulating equipment in the liquid line also sets the flow and is adjusted continuously from electronic readings to maintain desired process conditions.

Working Example

When the device of **Figure 3** is used in its simplest form (measuring gas flow only), the electrical power required (E) is equal to the product of specific heat (S.H.) times the temperature change produced by the device (ΔT) times the gas flow rate (G)

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Or:

$$E \text{ (Watts)} = S.H. \text{ (Joules per gram per } °C\text{)}$$
$$\times \Delta T \text{ (}°\text{Centigrade)}$$
$$\times G \text{ (Grams per second)}$$

Or: $E = S.H. \times \Delta T \times G$

For argon gas (commonly used), $SH = 0.523$

Any ΔT can be chosen which doesn't damage apparatus or gas. A convenient ΔT may be 85° C.

In that case,

$$E = 0.523 \times 85 \times G = 44.455 \, G$$

Or, $G = .0225 \, E$

Thus, measurement of the input wattage is a direct indication of the gas flow.

It may be more convenient to select an output temperature before hand as part of the process. The selection would be based on one not damaging to apparatus or gas, maximization of measurement signal, minimization of delivery tube heating etc. While such selection sounds complicated, it is instead a means of optimizing process conditions to reduce complications.

In that case,

$$E = 0.523 \, (T_3 - T_1) \times G$$

If $T_3 = 85°$ C, and $T_1 = 25°$ C, and the desired process gas flow is 0.0545 grams per second of argon,

$$E = 0.523 \, (85 - 25) \times 0.0545$$
$$= 1.71 \text{ watts}$$

The volume of the argon flow depends on temperature and pressure. At 25° C and atmospheric pressure, 0.0545 grams per second represents 2.0 liters of gas per minute.

Now suppose a liquid is also included in the flow. The liquid, in addition, is to be converted (evaporated or vaporized) to a gas. Typically, the intention is to deliver the liquid in gaseous form to the process area. This means that, after evaporation, conditions must be maintained to avoid condensation. All liquids have strict physical properties that govern their transition to gases and also re-condensation.

If the liquid is water, for example, it will evaporate or condense in accordance with its fundamental vapor pressure properties. To evaporate water, it is not necessary to "boil" it. The air we breathe contains water which is obviously not "boiling" (100° C) and which is indicated by humidity and dew-point references. The term "boiling point" means the temperature at which surrounding air, at atmospheric pressure, can be 100% replaced by gaseous water. The boiling

point of water (or any other liquid) is thus the temperature at which the vapor pressure equals the ambient pressure.

The above measurement temperature, T_3, will indicate the maximum water concentration, or vapor pressure, leaving the outlet 316 in **Figure 3**. As mentioned, T_3 can be whatever temperature is suited to the purpose intended. If T_3 is 85°, the vapor pressure of water at this temperature is 433.6 millimeters of mercury (mm Hg). If the pressure in the flow channel 315 is at atmospheric (760 mm Hg), this means the maximum concentration of water vapor in the outlet 316 of **Figure 3** is:

$$\frac{433.6}{760} = 0.571 = 57.1\%$$

If the pressure in the flow channel 315 is regulated to 433.6 mmHg, the water vapor content can now be 100%. However, this would be equivalent to 100% humidity, meaning condensation would occur at the slightest provocation. Process control may well dictate that 90% vapor pressure (390.24 mmHg) be a practical maximum to stay away from unstable conditions. In that case, the water vapor in the outlet of **Figure 3** is either 100% at a pressure of 390.24 mm Hg or 51.3%

$$(\frac{390.24}{760} \times 100 = 51.3\%)$$

at atmospheric pressure, mixed with argon. The argon is serving as a "carrier gas". Evaporated liquid vapors always have limitations regarding temperature and pressure. Adding a non-condensable inert gas like argon allows choice of pressure and temperature without violating vapor pressure limitations.

At 85 °C, and 760 mm Hg, the density of argon gas is 1.360 grams per liter (gm/l). At the same conditions, water vapor density is 0.6135 gm/l. If the argon process gas flow chosen is 0.0545 grams per second, this corresponds to 0.040 liters per second (2.40 liters per minute) at 85 °C and 760 mm Hg. The designated 51.3% water vapor in the outlet flow is by volume, not by weight. The argon is thus 48.7% of the total, at 0.040 liters per second, and the water is 0.042 liters per second or 0.0258 grams per second (1.55 grams per minute).

A typical process can be one cubic centimeter of liquid water per minute (one gram per minute) which would be 0.017 grams per second (0.027 liters per second). In that case the mixture leaving the outlet 316 of **Figure 3** is 40.3% water vapor which is even less likely to have condensation instability problems than the 51.3% maximum mentioned earlier.

The electrical power, which is an indication of these desired flows, can now be determined. The water is heated from T_2 to T_3. The specific heat of water is 4.186 Joules per gram per degree C. Evaporation of the water involves heat of vaporization, which for water at 85°C, is 2294.3 Joules per gram. Thus the energies involved are:

Argon: $E_1 = 0.523 (85-T_1)$ x .0545 = 1.7 watts (T_1=25°C)

Water: $E_2 = 4.186 (85-T_2)$ x .017 = 4.27 watts ($T_2 = 25$°C)

$E_3 = 2294.3$ x .017   = 39.0 watts

Total: 44.97 watts

Therefore, a measurement of 44.97 watts indicates a flow of 0.017 gm per second of water and 0.0545 gm per second of argon.

The numerous subtle details described are due to process and materials, and not due to the complexity of the device of **Figure 3**. Traditional flow measurement techniques frequently have difficulty keeping up with these subtleties for optimal process control.

The device of **Figure 3** is a comparatively simple and cost-effective way to cope with the complexities of modern day processing.

The above example illustrates argon and water. But gas mixtures and liquid mixtures can also be measured and fully vaporized to serve the needs of modern industry.

The flow measurement system of **Figure 3** has numerous advantages over conventional methods of mass flow measurement shown in **Figure 1** and **Figure 2** as follows.

1. It works with gases or liquids.
2. It works with mixed liquids.
3. It's much simpler than one or multiple bubblers.
4. It's much smaller than bubblers.
5. The internal coaxial heat source keeps much better track of the thermal effects that are the basis of the flow measurement.
6. Without capillary or bypass, all fluid flow is measured, thus eliminating by-pass errors.
7. Temperature measurement is internal for improved accuracy.
8. The bubbler pressure sensitivity is eliminated.
9. The bubbler requirement for exact (and unstable) gas-vapor saturation is eliminated.
10. Temperature controlled refill is eliminated.

While the invention has been described in connection with what are presently considered to be the most practical and preferred embodiments, it is to be understood that the invention is not

limited to the disclosed embodiments, but rather is intended to cover various modifications and equivalent arrangements which are included within the spirit and scope of the following claims.